## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants

Hitoshi Asahi et al.

Serial No.

10/588,837

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For

STEEL PLATE OR STEEL PIPE WITH SMALL OCCURRENCE OF BAUSCHINGER EFFECT AND

METHODS OF PRODUCTION OF SAME

Examiner

Velasquez, Vanessa T.

Art Unit

1733

Confirmation No.

4507

## Mail Stop AMENDMENT

Commissioner for Patents P.O. Box 1450

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# **DECLARATION OF HITOSHI ASAHI UNDER 37 C.F.R. § 1.132**

I, Hitoshi ASAHI, a citizen of Japan, declare as follows:

#### **Background**

- 1. I am a co-inventor of the invention(s) disclosed and claimed in the above-identified patent application.
- 2. I am an employee of Nippon Steel Corporation, Tokyo, Japan. Nippon Steel Corporation, Tokyo, Japan is the assignee of the above-identified patent application.
- 3. I earned my Master Degree from Osaka University Graduate School, Japan in March 1981. I earned my Doctor of Engineering from Osaka University, Japan in March 1994.
- 4. I have been employed by Nippon Steel Corporation, Tokyo, Japan, since April 1981. My work at Nippon Steel Corporation includes pipe and tube research and development. I am currently Chief Researcher for pipe and tube research in the Steel Research Laboratories of Nippon Steel Corporation.
  - 5. I read and understand both the English language and the Japanese language.
- 6. I have read and understand the specification, drawings, and pending claims 7 to 12 of the above-identified patent application. I have read and understand the Final Office

Action (the Final Office Action) dated September 9, 2009, the Advisory Action (the Advisory Action) dated February 18, 2010, and the Office Action (the Office Action) dated December 22, 2010, particularly the comments in the Office Action concerning my Declaration under U.S.C. § 1.132 dated July 28, 2010. I have read and understand the prior art of record cited against the above-identified patent application:

Japan No. 10-176239 to Kashima et al. (Kashima), which I read in the original Japanese;

Bates et al. "Quenching of Steel," Vol. 4, Heat Treating, ASM Handbooks On Line, ASTM International, 2002, (49 pages total), particularly pages 1 to 5 of 49, Figure 42(b) and Figure 43; and

JP 2003-096545 to Kami et al. (Kami), which I also read in the original Japanese.

- 7. In the Office Action, claims 7 to 12 are rejected under 35 U.S.C. § 103(a), as being unpatentable over Kashima taken alone. In the Final Office Action, the claims were rejected over Kashima in combination with Bates.
- 8. Bates is not cited in the rejection of the claims in the Office Action. However, in acknowledging my previous Rule 132 Declaration, dated July 28, 2010, the Office Action states that my previous Rule 132 Declaration was "insufficient" to overcome the rejection in the Final Office Action of claims 7 to 12 under 35 U.S.C. § 103(a) over Kashima in view of Bates.
- 9. The Office Action states that the word "small" in the recitation in the claims of a "small occurrence of the Bauschinger effect" is not defined. The Office Action states that "small" is a relative term, and without establishing a standard for the term "small," it is unclear how data can be compared to demonstrate a significant difference between the presently claimed steel pipe and the pipe disclosed in the cited prior art references.
- 10. The Office Action also states that my previous Rule 132 Declaration does not calculate the proportional limit ratios of the tested steels to allow a comparison of the tested steels and the claimed range.

## **The Claimed Invention**

11. In response to those statements in the Office Action, the claims have been amended to delete "small occurrence of the Bauschinger effect," and to add the recitation that a steel pipe, formed from a plate of the steel base material, heated at the austenite-ferrite dual-phase temperature region and then quenched, where the heating and quenching are after

the plate of the steel base material is shaped into the pipe, has a ratio of the proportional limit of the compression stress-strain curve in the circumferential direction before and after expansion of at least 0.7.

- 12. Therefore, as recited in claim 7, the presently claimed invention is directed to steel pipe formed from a plate of a steel base material. The steel base material comprises, by mass percent, C: 0.03 to 0.30 percent, Si: 0.01 to 0.8 percent, Mn: 0.3 to 2.5 percent, P: 0.03 percent or less, S: 0.01 percent or less, Al: 0.001 to 0.1 percent, N: 0.01 percent or less, and a balance of iron and unavoidable impurities, and has a dual-phase structure substantially comprising ferrite structure and fine martensite dispersed at the ferrite grain boundaries.
- 13. After a plate of the steel base material is shaped into a steel pipe, the steel pipe of the invention is heated at the austenite-ferrite dual-phase temperature region, and then quenched. The heating at the austenite-ferrite dual-phase temperature region in combination with the claimed quenching provides steel pipe having a ratio of the proportional limit of a compression stress-strain curve of the pipe in the circumferential direction before and after expansion of the steel pipe of at least 0.7.
- 14. Thus, the claimed invention provides a steel pipe for use in oil wells or gas wells that exhibits a drop in compression strength in the circumferential direction after the steel pipe has been expanded in the circumferential direction that is less than the drop that occurs in prior art oil and gas well pipe. Those skilled in the art recognize that the presently claimed steel pipe has a Bauschinger effect that is less than that of prior art pipe, i.e., a "small occurrence of the Bauschinger effect." See the present specification, page 1, lines 10 to 14.
- 15. As discussed in my previous Rule 132 Declaration, the phenomenon known as the "Bauschinger effect" is well known to those skilled in the art. In this phenomenon, if tensile stress is applied to a steel article in a given direction to cause plastic deformation, and then compressive stress is applied in the opposite direction, tensile stress reapplied in the original direction causes plastic deformation to occur at a lower stress than the original yield strength. Specification, page 1, lines 24 to 29. The combination of the heating and quenching of the steel pipe of the invention minimizes the Bauschinger effect in the circumferential direction when the steel pipe has been expanded (tensile stress) in the circumferential direction.

- steel pipe used in oil or gas wells is expanded 10 to 30 percent after drilling. Expandable tubular technology reduces drilling costs by expanding the steel pipe that has been inserted into the well after drilling. The expansion of the steel pipe introduces high tensile stress in the circumferential direction. The ground introduces a high circumferential compressive stress in the opposite direction. Therefore, the Bauschinger effect on the steel pipe in such situations has become an issue. See the present specification, page 2, lines 29 to 36. The invention of the present application is directed to providing a steel pipe that has an occurrence of the Bauschinger effect that is less than that occurring in prior art pipe. Such a pipe is obtained in accordance with the present invention when the steel pipe has a ratio of the proportional limit of the compression stress-strain curve in the circumferential direction before and after expansion of at least 0.7.
- 17. In the present invention, a steel pipe with an occurrence of the Bauschinger effect that is less than that of prior art pipe is fabricated from a steel plate comprising a steel base material containing, by mass percent, C: 0.03 to 0.30 percent, Si: 0.01 to 0.8 percent, Mn: 0.3 to 2.5 percent, P: 0.03 percent or less, S: 0.01 percent or less, Al: 0.001 to 0.1 percent, N: 0.01 percent or less, and a balance of iron and unavoidable impurities. The steel base material has a dual-phase structure substantially comprising ferrite structure and fine martensite dispersed at the ferrite grain boundaries.
- 18. Again, after the plate of the steel base material is shaped into the steel pipe, the steel pipe is heated at the austenite-ferrite dual-phase temperature region and then quenched, and the ratio of proportional limit of the compression stress-strain curve in the circumferential direction of the steel pipe before expansion of the steel pipe and after expansion of the steel pipe strain is at least 0.7 m where:

The ratio of the proportional limit is (PL-a)/(PL-b), where (PL-a) is the proportional limit yield strength after expansion of the steel pipe, and (PL-b) is the proportional limit yield strength before expansion of the steel pipe using a 0.05 percent offset yield strength.

19. The ratio of the proportional limit, (PL-a)/(PL-b), is called the "Bauschinger effect ratio." With a greater value of the ratio of the proportional limit, (PL-a)/(PL-b), the occurrence of the Bauschinger effect is less. See, the present specification, page 8, lines 5 to 8. Experimentally obtained values of the proportional limit ratio, i.e., the Bauschinger effect ratio, are provided in Table 4 of the present specification.

20. The steel pipe of the present invention, having an occurrence of the Bauschinger effect that is less than that of prior art steel pipe, is a steel pipe wherein, <u>after</u> a steel plate is shaped into the steel pipe, the steel pipe is heated at the austenite-ferrite dual-phase temperature region, and then quenched. There is no prior art disclosure of a steel pipe formed in the manner used to produce the steel pipe of the invention.

## The Prior Art

- 21. As I stated above, I have reviewed the Kashima reference in the original Japanese language. Kashima discloses a steel plate for use in forming steel pipe that may be similar in composition to the steel base material of the present invention. However, Kashima does not disclose or suggest that after a steel plate is shaped into a steel pipe, the steel pipe should be heated at the austenite-ferrite dual-phase temperature region and then quenched to obtain a ratio of the proportional limit of the compression stress-strain curve in the circumferential direction before and after expansion of at least 0.7, as presently claimed.
- 22. Bates was cited in the Final Office Action for the disclosure of water as a convenient and pollution-free means to quench steel and that water is capable of creating cooling rates within the range taught by Kashima. However, Bates does not disclose or suggest that the disclosed steel pipe should be heated at the austenite-ferrite dual-phase temperature region and then quenched after a steel plate is shaped into the disclosed steel pipe to obtain a ratio of the proportional limit of the compression stress-strain curve in the circumferential direction before and after expansion of at least 0.7, as presently claimed.
- 23. Therefore, Kashima and Bates taken alone or together do not disclose or suggest that <u>after</u> a steel plate is shaped into a steel pipe, the steel pipe should be heated at the austenite-ferrite dual-phase temperature region and then quenched, as is the steel pipe of the invention to obtain a ratio of the proportional limit of the compression stress-strain curve in the circumferential direction before and after expansion of at least 0.7.
- 24. The steel pipe product of the present invention, which has a ratio of the proportional limit of a compression stress-strain curve of the pipe in the circumferential direction before and after expansion of the steel pipe of at least 0.7, has a heat history that neither Kashima nor Bates disclose or suggest. The steel pipe of the present invention is significantly different from the steel pipe that one of ordinary skill in the art would obtain from the disclosures of Kashima and Bates.

- 25. As with Kashima, I have reviewed the Kami reference in the original Japanese language. Kami discloses a steel pipe that may be formed from a steel similar in composition to the steel base material used to make the steel pipe of the present invention. However, Kashima does not disclose or suggest that <u>after</u> a steel plate is shaped into a steel pipe, the steel pipe should be heated at the austenite-ferrite dual-phase temperature region and then quenched, as is the steel pipe of the invention to obtain a ratio of the proportional limit of the compression stress-strain curve in the circumferential direction before and after expansion of at least 0.7, as presently claimed.
- 26. The Office Action cites paragraphs [0013], [0016], and [0030] of Kami for disclosing heating a pipe to 650° to 850°C and subsequently cooling the pipe, where the Office Action states that cooling means quenching. However, as well known to those skilled in the steel making art, most disclosures of cooling are not disclosures of quenching. Cooling typically occurs slowly over an extended period of time. In contrast, quenching is performed rapidly, and requires rapid cooling, such as that obtained by spraying the heated steel with water or oil, or immersing the heated steel in water or oil. Quenching results in a very rapid decrease in the temperature of the heated steel.
- 27. An example of cooling that is not quenching is provided by the original Japanese disclosure of Kami in paragraph [0013] cited by the Office Action. In paragraph [0013], Kami discloses that an steel tube is heated to 650° to 850°C, subjected to 50 percent diaphragm rolling, and then cooled to 600°C at an average cooling rate of 2.0°C per second. One of ordinary skill in the art will understand that an average cooling rate of 2.0°C per second is far too slow to be considered quenching. A typical average cooling rate for quenching is typically 30°C per second, and a minimum cooling rate for quenching is about 10°C per second.
- 28. In paragraphs [0016] and [0030] Kami discloses cooling heated pipe in various steps of the process disclosed in that reference. However, the cooling disclosed in paragraphs [0016] and [0030] of Kami is not quenching.
- 29. Therefore, Kami does not disclose or suggest that <u>after</u> a steel plate is shaped into a steel pipe, the steel pipe should be heated at the austenite-ferrite dual-phase temperature region and then quenched, as is the steel pipe of the present invention. The steel pipe disclosed by Kami will have a ratio of the proportional limit of a compression

stress-strain curve of the pipe in the circumferential direction before and after expansion of the steel pipe that is significantly less than the presently claimed 0.7.

30. Kami does not disclose or suggest a steel pipe that has the heat history of the presently claimed steel pipe. The steel pipe of the present invention is significantly different from the steel pipe that one of ordinary skill in the art would obtain from the disclosure of Kami.

# **Comparative Tests**

- 31. Figures A, B, and C, which were submitted with my previous Rule 132 Declaration, are attached.
- a. Figure A provides a stress-strain diagram for a stress-strain test of a steel pipe in accordance with the present invention.
- b. Figure B provides a stress-strain diagram for a stress-strain test for a steel plate having a steel composition in accordance with the present invention, Kashima, and Kami.
- c. Figure C provides a stress-strain diagram for a stress-strain test for a steel pipe that has not been heated and quenched, such as the steel pipes disclosed by Kashima and Kami.
- d. As stated in my previous Rule 132 Declaration, the X-axis, i.e., the strain axis, in each of the diagrams illustrated in Figures A, B, and C represents the same units. In Figure A, the strain axis is expressed as percent. In Figures B and C, the strain axis is expressed as a non-dimensional decimal. It is readily apparent that the X-axis or strain axis of Figures A, B, and C represent the same units.
- 32. A steel plate was produced having the following composition in mass percent:
  C: 0.086 percent; Si: 0.21 percent; Mn: 1.19 percent; P: 0.018 percent; S: 0.006 percent;
  Al: 0.03 percent; N: 0.0035 percent, and a balance of iron and unavoidable impurities;

The steel plate was produced by heating a slab at 1,230°C; hot rolling at a finish temperature of 870°C; cooling at a cooling rate of 20°/second; then coiling at a coiling temperature of about 550°C; and

Then it was determined that the steel base material of the steel plate had a dual phase structure substantially comprising a ferrite structure and fine martensite dispersed at the ferrite grain boundaries. The fine martensite had an area ratio of 15 percent.

- 33. The steel plate had a composition substantially the same as the steel plates used to obtain the results provided in the examples of the present specification.
- 34. A steel pipe of the invention was fabricated from the steel plate discussed above. After the steel pipe was fabricated from the steel plate:

The steel pipe was heated at the austenite-ferrite dual-phase temperature region; and The heated steel pipe was then quenched.

- 35. A test piece of the steel pipe of the invention was subjected to a stress-strain test. Figure A provides the results of the stress-strain test of the steel pipe of the invention, which was heated and quenched in accordance with the present invention. Figure A shows that after a steel plate of the present invention is shaped into a pipe, then heated at the austenite-ferrite dual phase temperature region, and then quenched, the resulting steel pipe of the invention absorbs additional stress from a load after reaching the yield point stress. That is, a steel pipe formed from the steel plate of the invention that is heated and quenched in accordance with the invention exhibits plastic deformation or working.
- 36. As can be seen in Figure A, the yield point stress of the test piece of the steel pipe in accordance with the invention is about 400 MPa. That is, the steel pipe of the invention begins to yield to the stress applied to the pipe when the stress is about 400 MPa. However, the steel pipe of the invention continues to absorb stress until the stress exceeds 600 MPa. Failure of the test piece of the steel pipe in accordance with the invention does not begin to occur until the strain on the pipe is greater than 15 percent.
- 37. Thus, as I stated in by previous Rule 132 Declaration, Figure A shows that the steel pipe of the present invention, which has been subjected to a specific heat treatment and then quenching after the steel plate is shaped into the steel pipe, does not begin to fracture when the stress reaches the yield point stress. Instead, the steel pipe of the present invention exhibits plastic working upon reaching the yield point stress, and continues to absorb stress as the stress increases.
- 38. In addition, the proportional limit ratio of the steel pipe of the invention used to obtain the results illustrated in Figure A will be at least 0.7, as presently claimed. The proportional limit ratio of that steel pipe must be at least 0.7, as that steel pipe had a composition that was substantially the same as the steel pipes of the invention used to provide the examples of the present specification, and was formed from a steel plate in the same manner as the steel pipes of the invention. As all of the exemplified steel pipes of the

invention have a proportional limit ratio of at least 0.7, those skilled in the art will understand that the steel pipe of the invention of Figure A must also have a proportional limit ratio of at least 0.7. Contrary to the statement in the Office Action that my previous Rule 132 Declaration does not provide a calculate proportional limit ratio for the steel pipe of Figure A, the value of the proportional limit ratio of that pipe must be at least 0.7. A pipe made from the same material in the same process must have the same properties.

- 39. Figure B provides stress-strain diagram for the steel plate used to form the steel pipe of the invention that provided the results of the stress-strain test illustrated in Figure A. That steel plate has the composition of the steel plate of the present invention and of steel plate used in the prior art, such as that used to form the pipe disclosed in Kashima and Kami. A test piece of that steel plate was subjected to a stress-strain test. Figure B shows that the tested steel plate absorbs additional stress from a load after reaching the yield point stress, and, thus, exhibits plastic deformation or working. The steel plate has a yield point stress of about 400 MPa, and continues to absorb stress until the stress approaches 500 MPa at a strain of about 10 to 15 percent.
- 40. Figure C provides the results of a stress-strain test of a the steel pipe that was not heated and quenched in accordance with the present invention. The steel pipe was fabricated from the steel plate disclosed above in substantially the same manner as the steel pipe discussed above. Thus, the steel pipe subjected to the stress-strain test of Figure C is a prior art pipe, such as the pipe disclosed by Kashima and Kami, that was not both heat treated and then quenched after the steel pipe was fabricated from the steel plate. The steel pipe will be referred to as a prior art steel pipe in this Rule 132 Declaration, as the prior art steel pipe has not been heat treated and then quenched after the steel plate is shaped into the steel pipe.
- 41. To obtain the results shown in Figure C, a test piece of the prior art steel pipe was subjected to a stress-strain test substantially the same as the stress-strain test of the test piece of the steel pipe test piece in accordance with the present invention described above. Figure C shows that the prior art steel pipe, which was not heat treated and then quenched after being shaped into a pipe, does not absorb additional stress from a load after reaching the yield point stress, and, thus, does not exhibit plastic working or plastic deformation.
- 42. In contrast to the steel pipe of the invention, the prior art steel pipe begins to fracture immediately after reaching the yield point stress. As the prior art steel pipe fractures

immediately after reaching the yield point stress, it is not possible to calculate a proportional limit ratio for the prior art steel pipe.

- 43. In contrast to the steel pipe of the invention, the prior art steel pipe does not exhibit any plastic working after reaching the yield point stress. At a yield point stress of a little more than 500 MPa and a strain of about 1 percent, the prior art pipe fails, and does not absorb any additional stress. Figure C shows that the stress absorbed by the prior art steel pipe reaches a maximum at about the yield point stress, and then fails. The prior art steel pipe is unable to absorb any additional stress, as does the steel pipe of the invention, which absorbed over 600 MPa of stress at a strain of over 15 percent.
- 44. As discussed above, the Office Action states that my previous Rule 132 Declaration does not establish that the results of the test discussed above are significant because the proportional limit ratio of the steel pipe of the invention and that of the prior art steel pipe are not calculated. However, those skilled in the art will understand from the present specification that the proportional limit ratio of the steel pipe of the invention used to obtain Figure A must be at least 0.7, and that a proportional limit ratio for the prior art steel pipe cannot be calculated. Thus, the prior art steel pipe does not have a proportional limit ratio of at least 0.7, as presently claimed.
- 45. As a result of having the proportional limit ratio of at least 0.7, the steel pipe in accordance with the present invention continues to absorb stress after reaching a yield point stress of about 400 MPa, such that the steel pipe in accordance with the invention exhibits plastic deformation or working to a maximum stress of over 600 MPa at a strain of over 15 percent. Thus, steel pipe in accordance with the present invention exhibits the ability to absorb stress at a stress that was over 50 percent higher then the yield point stress. Such an ability to absorb stress beyond the yield point stress is a clear indication of a relatively large proportional limit ratio.
- 46. In contrast to the steel pipe in accordance with the present invention, as a result of having the proportional limit ratio that is not at least 0.7, the prior art steel pipe failed to absorb any appreciable stress above the yield point stress of about 500 MPa, and immediately failed at the yield point stress with a strain of only about 1 percent. Such an inability to absorb stress beyond the yield point stress is a clear indication that the presently claimed steel pipe is significantly different from and clearly superior to the prior art steel pipe.

47. Similar results are illustrated in the Examples and Figures provided in the present specification.

## Conclusion

- 48. Thus, Figure A illustrates the results of a stress-strain test of a steel pipe in accordance with the present invention having a ratio of the proportional limit of the compression stress-strain curve in the circumferential direction before and after expansion of at least 0.7, as presently claimed. Figure C illustrates the results of a stress-strain test of a steel pipe that has not been both heated and quenched in the manner of the steel pipe in accordance with the present invention, and, thus, does not have a ratio of the proportional limit of the compression stress-strain curve in the circumferential direction before and after expansion of at least 0.7. A comparison of Figures A and C shows that the two steel pipes, although formed from steel plates having substantially similar compositions, but having different heat histories, exhibit very different stress-strain characteristics.
- 49. An analysis of Figures A and C demonstrates that a steel pipe in accordance with the present invention is able to absorb stress at a stress that was over 50 percent higher then the yield point stress. In contrast, a prior art steel pipe fails at the yield point, and cannot absorb any additional stress. Thus, the steel pipe in accordance with the invention has a Bauschinger effect that is less than the Bauschinger effect of the prior art steel, which differs from the steel pipe in accordance with the present invention only in that the prior art steel pipe lacks the heat treatment and quenching of the steel pipe in accordance with the steel pipe of the invention. A prior art pipe that has been expanded, i.e., subjected to circumferential tensile stress, in applications such as oil and gas wells will fail at a lower stress than will an expanded steel pipe in accordance with the present invention formed from the same type of steel plate having the same dimensions.

#### **Declaration**

50. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

Hitoshi ASAHI

June 8, 2011

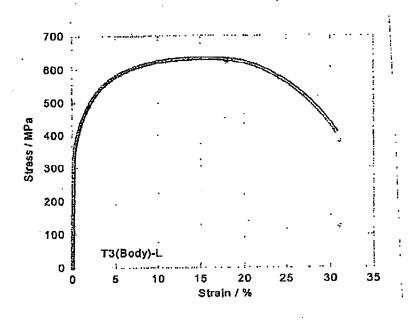


Fig. A

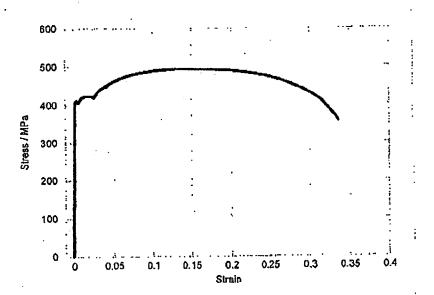


Fig. B

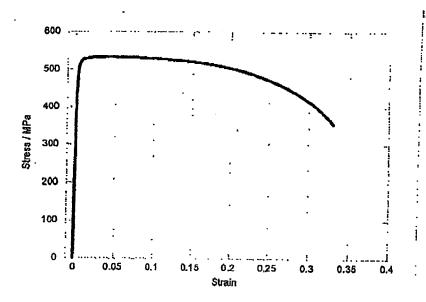


Fig. C